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F031/F004

AUTHOR: Liang, Shu-ch'üan (2733/2885/2938) and Hung, Shui-chien (3163/3055/4105)

TITLE: On the separation and determination of scandium and thorium; III. With tetrachlorophthalic acid as reagent

PERIODICAL: Hua Hsüeh Hsüeh Pao, v. 28, no. 3, 1962, 139-147

TEXT: The authors carried out experiments on the separation and determination of scandium and thorium using tetrachlorophthalic acid as reagent. Solutions of 0.3% and 0.1% tetrachlorophthalic acid manufactured by the Dr. Theodor Schuchardt Factory, Munich were used. The acid was recrystallized prior to use. Instruments used were the same as those of previous experiments. Experiments covered: 1. Relation between acidity and the quantitative precipitation of scandium. Put scandium nitrate solution containing 15.6 mg scandium oxide in a flask, dilute with water to 50 ml, add 100 ml of 0.3% tetrachlorophthalic acid, adjust to desired pH by 2N NH_4OH

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or 2N HCl, agitate and wait 2-3 hrs, filter and dry the precipitate at 110°C, then heat in 350°C oven and burn at 850°C for 1 hr. Results showed that at pH 1.0-1.2, the precipitate of Sc can be neglected while thorium can be precipitated and in pH 2.4-4.4, scandium can precipitate quantitatively. The precipitation of Sc will not be complete above pH 4.4. 2. Dose of precipitant. 2.5-3 moles of reagent is required to precipitate one gram atom of scandium. However, a higher dose (up to 7.8 moles) of precipitant will do no harm to the precipitation. 3. Precipitation range. The method is suitable for 1-102 mg of scandium oxide or 0.6-67 mg of Sc. 4. Thermolytic curves. Scandium and thorium tetrachlorophthalates were prepared under analytical conditions and subjected to thermolysis. The salts were heated in a drying oven for temperatures under 200°C or for those higher than 200°C in an electric muffle furnace. Temperature increase was 2°C/min at the beginning and, after 200°C, 5°C/min. Thermolytic curves for scandium and thorium tetrachlorophthates are similar in shape to two plateaus indicating respectively the formation of basic tetra-chlorophate salts and oxides. The plateaus for the Sc salt are (a) 80-150°C and (b) 670-960°C and those for the Th

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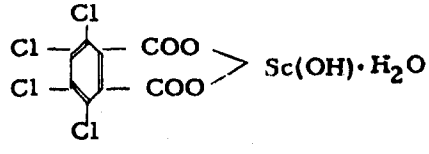
salt are (a) 80-150°C and (b) 720-960°C. The structural formula of the Sc salt was found to be



and experimental formula $C_6Cl_4(COO)_2Sc(OH)\cdot H_2O$. The Sc salt contains experimentally C, 25.21, 25.42; H, 0.74, 0.63; Cl, 36.73, 36.91; Sc, 11.60, 11.45 and the Th salt, $[C_8Cl_4O_4]_2Th\cdot 3H_2O$ contains Th, 25.97 and 25.96. 5. Effects of several kinds of salts on the precipitation of Sc. A large quantity (up to 4 gm) of ammonium salts in form of nitrate, obloride, and acetate have no effect on precipitation but sulphate ions do owing to their complexing with Sc (III). Though acetate ions also complex with Sc (III), the precipitation of scandium tetrachlorophthate will lower the concentration of acetate ions. Sodium and potassium ions will cause slightly positive errors. 6. Separation of Sc from other rare earths. Within pH 2.5-3.4, only a small

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quantity of light lanthamides will be precipitated. Erbium (III) and yttrium (III) precipitate quantitatively at pH 8.1-9.5 and pH 10 respectively. Ceric ions will not precipitate quantitatively at pH 0.5 however interference with determination of Sc and Th can be avoided by reducing them to cerous status. Experiments on the separation of Sc from light lanthamides (up to Sc : Ln = 1 : 96) and yttrium (up to Sc : Yt = 1 : 10.4) thru double precipitation with tetrachlorophthalic acid have been made successfully. 7. Procedures for separating and determining Sc and Th. Precipitate thorium salt at pH 1.0-1.1, treat, treat the precipitate according to L. Gordon's method, adjust to pH 30, treat once again, stir occasionally on water bath for 2 hours, then wash and cool, and filter with retentive paper. Treat scandium precipitate the same as thorium precipitate. Results show that scandium oxide and thorium oxide in weight ratio of 1:2.45 to 1:9.79, has an absolute error ranging from 0.1 to 0.3 mg for scandium oxide and 0.01 to 0.7 mg for thorium oxide. The authors thank Li I-yü (2621/0044/7183) for his participation in the experiments. There are 6 tables and 1 figure. References in English include: L. Gordon, C. H. Vanselow and H. H. Willard, Anal. Chem. 21, 13 3

(1949); R. C. Vickery, "The Chemistry of Yttrium and Scandium", P. 92, Pergamon, London, 1960; N. A. Lange, "Handbook of Chemistry", 8th ed., P. 1232, Handbook Publishers, Sandusky, Ohio, 1952.

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